MTL TR 92-27

THERMAL EXPANSION MEASUREMENTS OF POLYMER MATRIX COMPOSITES AND SYNTACTICS

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CERAMICS RESEARCH BRANCH

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ABSTRACT

The coefficients of thermal expansion (CTE) of various polymer matrix materials were measured using a thermal mechanical analyzer. The measurements include a set of standard materials used as calibration materials for the instrument. The data for the base polymers, syntactics, and E and S-2 glass-reinforced polyester matrix composites were obtained in the temperature range from -77°C to 120°C. The results for all of the polymeric materials, especially for the epoxy and epoxy syntactic (above 65°C), are nonlinear. The CTE of the syntactics were reduced by a factor of ~2 for the base resins. The data taken for the glass-reinforced composites show peaks and/or humps in the thermal expansion. These features are believed to be from the formation of voids within the plies of the composite laminate. The irregular behavior of the thermal expansion was minimized by thermal annealing under load conditions, whereas annealing in free-space does not significantly dampen this behavior. The thermal expansion of the material appears to be directly related to the fiber loading.

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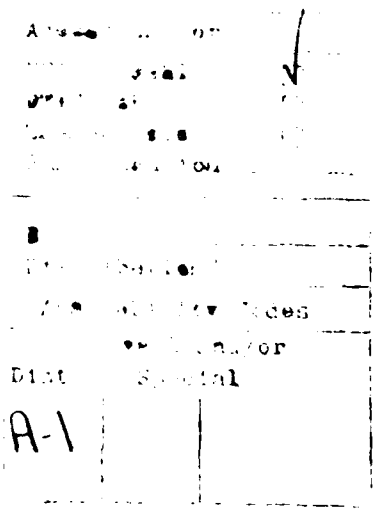

INTRODUCTION

In order to properly develop a composite material or structure which may be exposed to even moderate excursions in temperature, it is necessary to know the thermal behavior of the composite and its constituent materials. In addition, in order to bond these composites together for fabrication of dielectric skins or other applications, knowledge of the coefficients of thermal expansion (CTE) of the components is essential.

The relationship for the thermal expansion of a solid is:

$$\Delta L = \alpha L \Delta T \quad (1)$$

where α is in units (l/C^{o}), ΔL is the change in length (mm), length (mm), and ΔT is the temperature change. Equation 1 assumes a linear relationship with α being the slope of the line.

EXPERIMENTAL

The resins that were measured were 50.0 parts by weight (PBW) EPON® 828 (Shell Chemical) epoxy combined with 50.0 PBW EPON® V-40 polyamide curing agent (Shell Chemical) and Owens Corning (E-780) polyester combined 1 PBW MEKP and 0.1 PBW co-napthanate. The epoxy syntactic was fabricated using 52.0 PBW EPON® 828, 43.3 EPON V-40, and 4.6 PBW Grace Syntactics (Type S) microballoons. The polyester syntactic was 94.2 PBW polyester (E-780), 1 PBW MEKP, 0.1 PBW co-napthanate, and 4.3 PBW microballoons. The resins and syntactics were processed in air using the standard cure cycle for the resins. The S-2 glass and E-glass composites contained the following fabrics:

- JP Stevens (JPS) S-2 glass fabric, style 016781, an 8.8 oz. five harness satin weave, 55 picks by 57 ends with a JPS 09827 finish.
- A 5 x 5, S-2 glass with 24 oz. woven roving with an Owens Corning 463 finish.
- A 3 x 1, S-2 glass with 27 oz. woven roving with an Owens Corning 933 finish, nominally equivalent to JPS 09827 finish.
- A (0,90) 18 oz. stitched E-glass fabric.

The composites were fabricated by hand laminating the layers of fabric with wet polyester resin (Owens Corning E-780) and subsequently processing the composites using the standard vacuum bag cure cycle for this polyester-based material.

All of the samples were subsequently cut into 1/4" x 3/4" pieces. Flat parallel top and bottom sections are essential for accurate data acquisition since contact with the entire sample surface with the expansion probe is required.

The thermal expansion data was obtained using a Perkin Elmer, Series 7 Thermal Mechanical Analyzer. The experimental conditions consisted of: an applied load of 10 mN, a temperature ramp of $5^{o}C$/min to $40^{o}C$/min, and temperature ranges of $-77^{o}C$ to $120^{o}C$ for the polymers and $30^{o}C$ to $400^{o}C$ for the nonpolymeric materials used for standards. The best results were obtained by surrounding the sample chamber with liquid nitrogen thereby providing a thermal sink for consequently stabilizing the temperature ramp.

As a check on the calibration of the instrument, the CTEs of several standard materials were measured. The results are shown in Table 1. The percentage error is the difference between the average of two or more measurements and available literature values.[1]

Table 1. THE COEFFICIENTS OF THERMAL EXPANSION, α ($1/C^0$) FOR SOME STANDARD MATERIALS

Material	Measured ($1/C^0$)	Error (%)
Steel	11.4×10^{-6}	4
Silica	42.5×10^{-8}	8
Teflon	14.6×10^{-5}	Not Known
Aluminum	23.8×10^{-6}	4

The increased error for the silica sample is due to the measurement nearing the sensitivity limit of the instrument. The above results indicate that the results for unknown polymeric material based systems may, therefore, be accurate within ~4% since the order of magnitude is around 10^{-6}.

RESULTS AND DISCUSSION

As shown in Figures 1a and 1b, respectively, the thermal expansion data obtained for epoxy and epoxy syntactic are nonlinear. The figures show that the epoxy resin and syntactic have two distinct thermal expansion behaviors in this temperature range. The thermal expansion of the epoxy resin increases nonlinearly above 60°C, while the syntactic shows a similar increase above ~70°C. For the epoxy resin, the ratio of the thermal expansion found in the first temperature region (30°C to 60°C), to that of the second (60°C to 120°C) is 2.8. For the epoxy syntactic, the ratio of the thermal expansion found in the first region (30°C to 70°C) to that of the second region (70°C to 120°C) is 2.1. The epoxy resin thus appears to be more nonlinear in behavior than the syntactic. As shown in Figures 2a and 2b, similar behavior is observed for the polyester resin and the polyester syntactic. Table 2 summarizes the thermal expansion measurements obtained for the resins (polyester and epoxy and their syntactics).

Table 2. THE COEFFICIENTS OF THEMAL EXPANSION (CTE), α ($1/C^0$) FOR RESINS AND RESIN SYNTACTICS

Material	TEC ($1/C^0$)
Polyester	90.4×10^{-6}
Polyester Syntactic	45.9×10^{-6}
Epoxy	72.5×10^{-6}, 20.4×10^{-5}
Epoxy Snytactic	54.7×10^{-6}, 11.5×10^{-5}

1. Handbook for Physics and Chemistry. 55th Edition. R. C. Weast, ed., CRC Press, Cleveland, OH, 1975, p. D152.

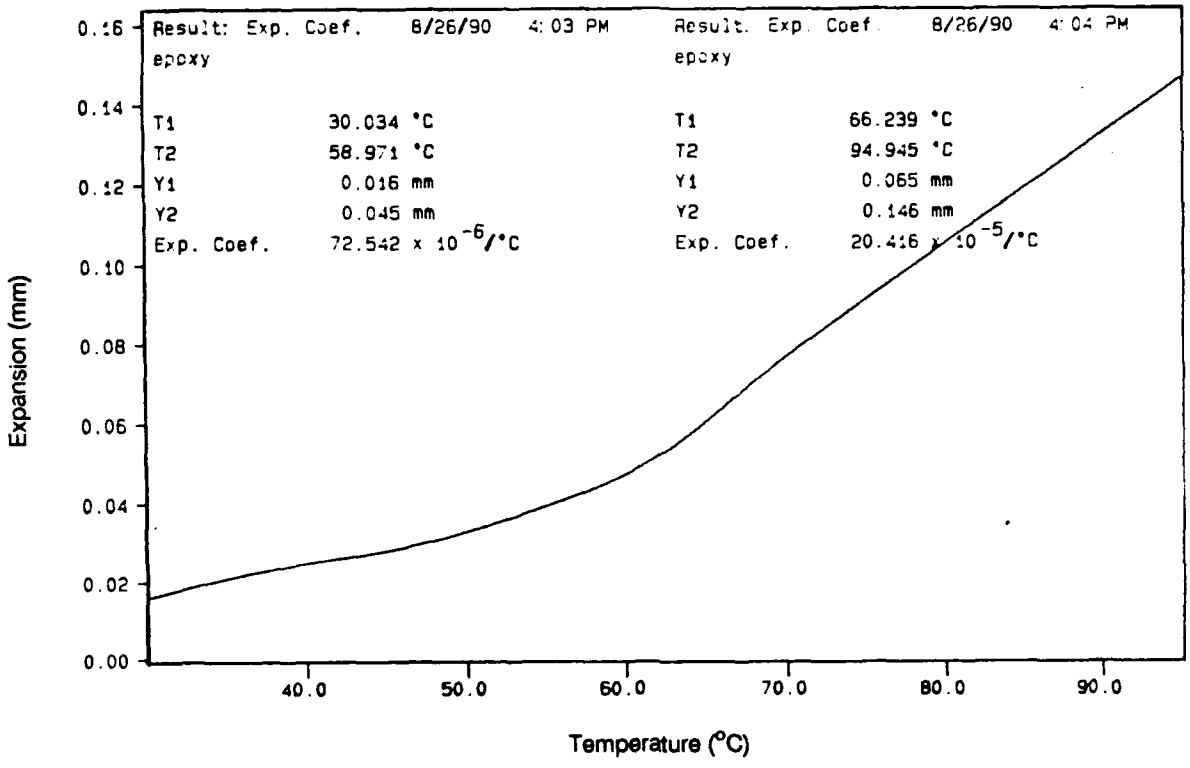

Figure 1a. The thermal expansion of epoxy resin.

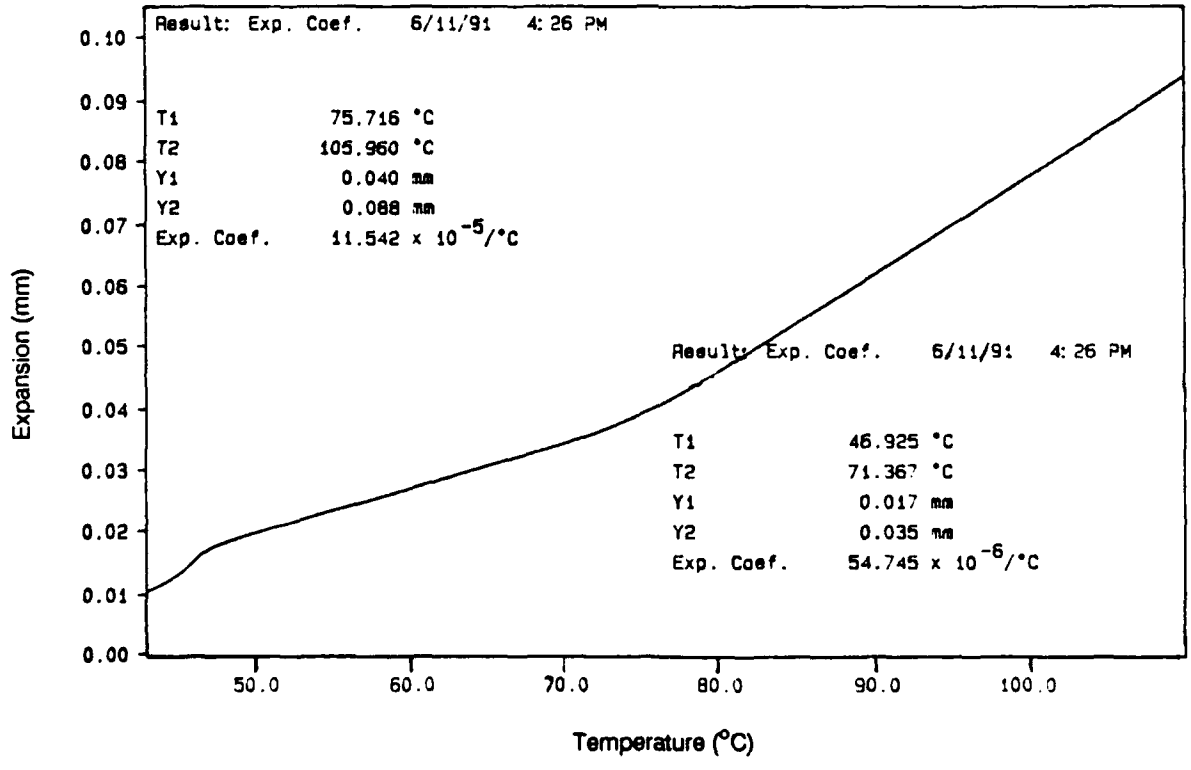

Figure 1b. The thermal expansion of epoxy syntactic.

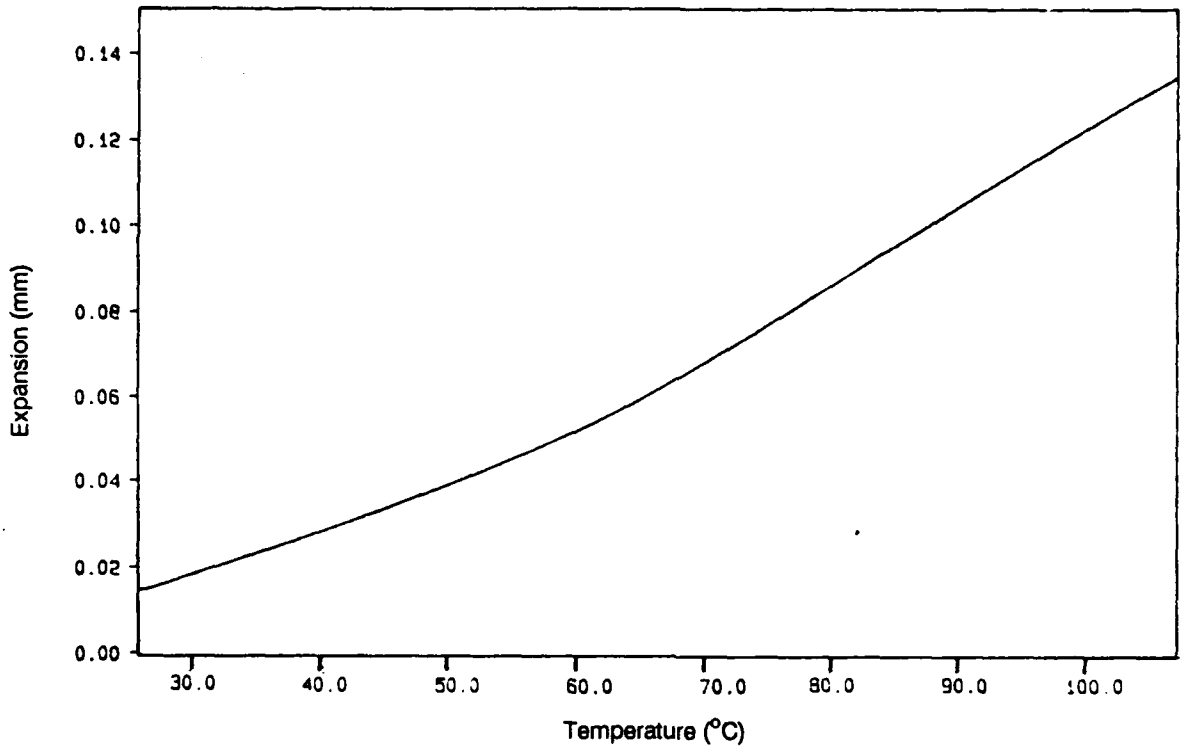

Figure 2a. The thermal expansion of polyester resin.

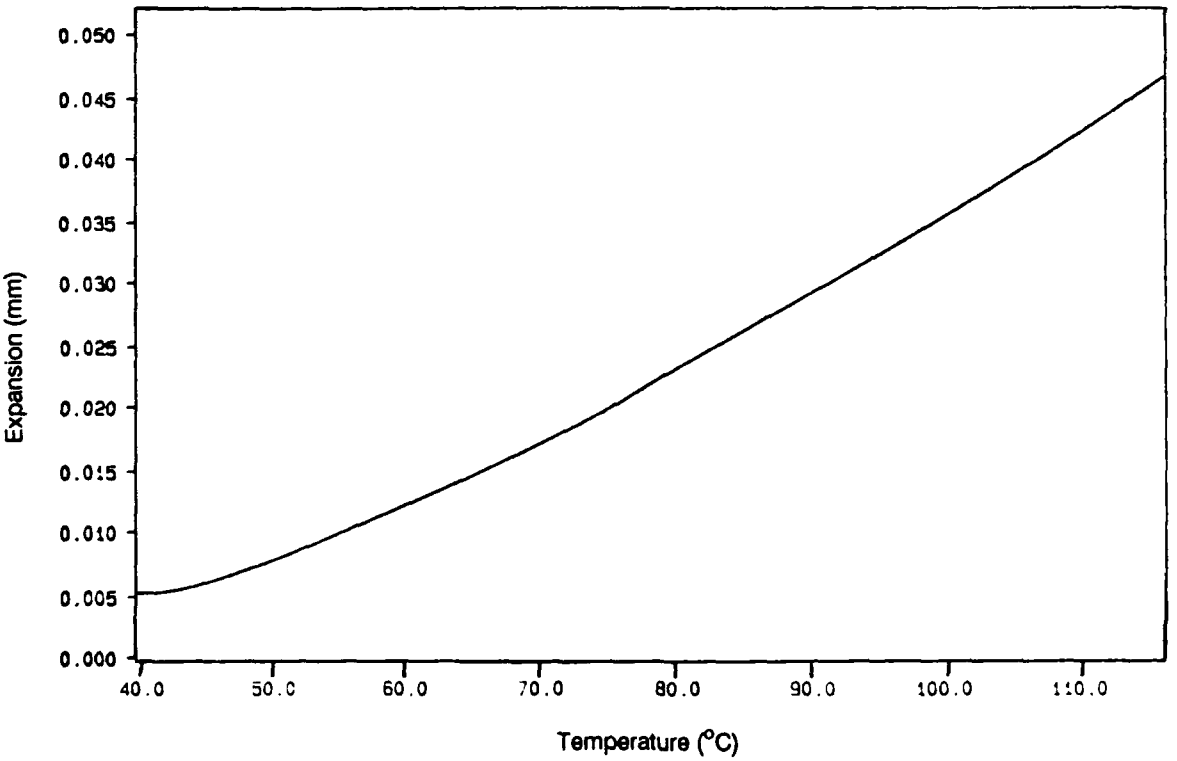

Figure 2b. The thermal expansion of polyester syntactic.

The expansion of the polyester syntactic is reduced by a factor of 2 when compared to the base polymer. The epoxy syntactic displays a reduction of 1.3 in the first region of the thermal expansion and a reduction of about 2 in the second region of expansion as compared to the epoxy resin. As observed, bonding the syntactics to the higher CTE base resins or to lower CTE glass-reinforced composites (to be discussed) at elevated temperatures may result in the fracturing of the syntactic.

Thermal expansion measurements were also obtained for composites fabricated from the glass fabrics listed above. The thermal expansion data from 30°C to 120°C for the S-2 glass (8.8 oz.) polyester resin matrix composite was taken both as parallel (see Figure 3a) and perpendicular (see Figure 3b) to the fibers. In addition, the thermal expansion was obtained for a composite fabricated from the same fabric that had been treated with polypyrrole (measured parallel to the fibers). As expected, the treatment does not alter the thermal expansion properties (see Figure 4). Figures 5a and 5b display the results obtained for the E-glass composite. Figures 6a and 6b show the data obtained for the S-2 glass 5 x 5 (24 oz.) composite. Figures 7a and 7b show the results for the S-2 glass 3 x 1 (27 oz.) composite. Table 3 summarizes the results obtained for the thermal expansions of the various composites examined which were measured both perpendicular and parallel to the laminate fibers. The values reported here reflect measurements obtained on samples which were annealed for various times in order to minimize error due to strain in the material.

Table 3. THE CTE, α ($1/C^0$) FOR THE COMPOSITE MATERIALS MEASURED PARALLEL AND PERPENDCULAR TO THE FIBERS. ALL TEXTILES ARE WITHIN A POLYESTER RESIN

Material	Perpendicular to Fibers ($1/C^0$)	Parallel to Fibers ($1/C^0$)
E-Glass	78.1×10^{-6}	15.0×10^{-6}
S-2, 5 x 5 (24 oz.)	28.7×10^{-6}	5.0×10^{-6}
		8.0×10^{-6} Polypyrrole Treated
S-2 (8.8 oz.)	41.7×10^{-6}	10.2×10^{-6}
S-2, 3 x 1 (27 oz.)	81.5×10^{-6}	14.9×10^{-6}

The E-glass and S-2, 3 x 1 (27 oz.) composites have CTEs which are similar in value and show a ratio of the CTE measured perpendicular over the parallel measurement of about 5.3. In contrast, the S-2, 5 x 5 (24 oz.) composite shows a ratio of 6 between the perpendicular and parallel measurement of the thermal expansion. The S-2 glass (8.8 oz.) composite shows a difference in the measurements taken parallel and perpendicular to the fibers of the fabric of about 4.8. The S-2 glass (8.8 oz.) composite (when measured perpendicular to the fabric) has a CTE about the same as the polyester syntactic, whereas the E-glass and the S-2 glass 3 x 1 (27 oz.) composites when measured perpendicular to the fibers have a CTE about the same as the polyester resin itself. This type of thermal behavior is expected since the expansion is directly related to the volume filling fraction, V_{RESIN}, Young's modulus (tensile modulus), E_{RESIN}, of the resin and inversely related to the volume filling, V_{FIBERS}, and Young's modulus, E_{FIBERS} of the fibers, as seen from the following expression[2]

$$\alpha_{COMPOSITE} = \alpha_{FIBER} \frac{5V_{RESIN} E_{RESIN} \alpha_{RESIN}}{V_{FIBER} E_{FIBER}} . \qquad (2)$$

2. Thermal Characterization of Polymeric Materials, R. Bruce Prime and E. A. Turi, ed.

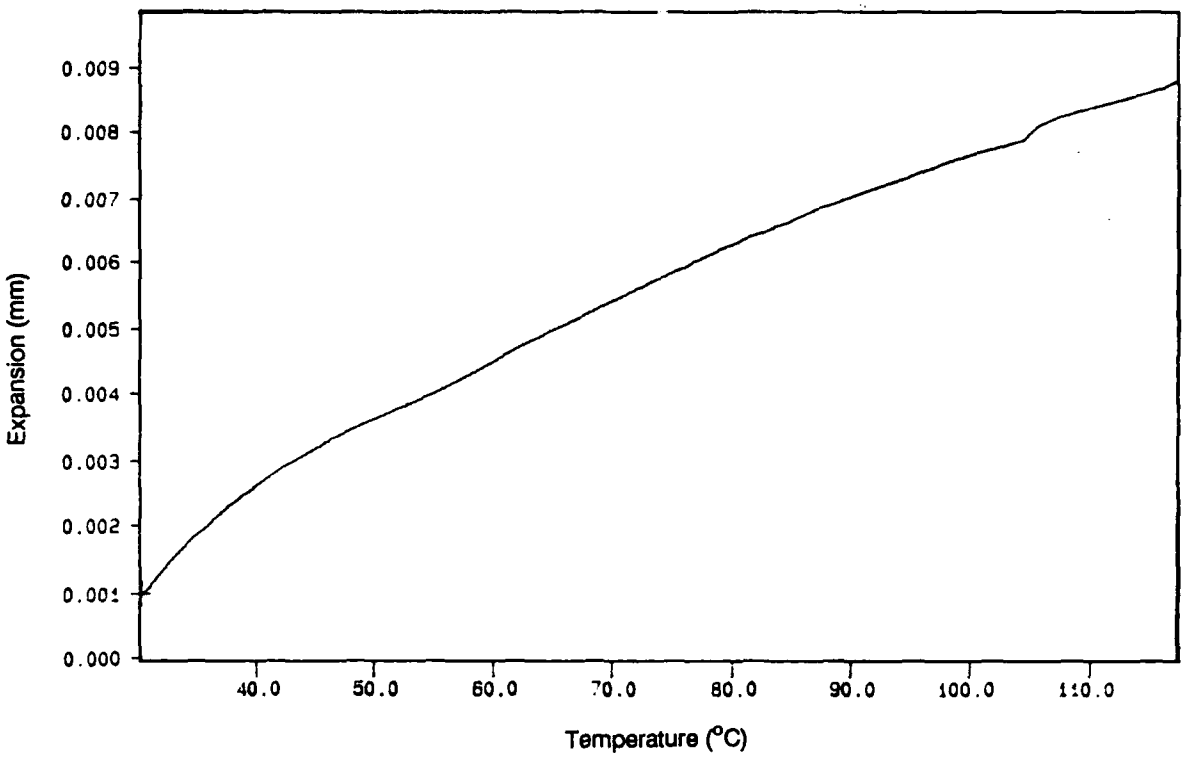

Figure 3a. The thermal expansion for a S-2 glass (8.8 oz.) polyester resin matrix composite parallel to the fibers.

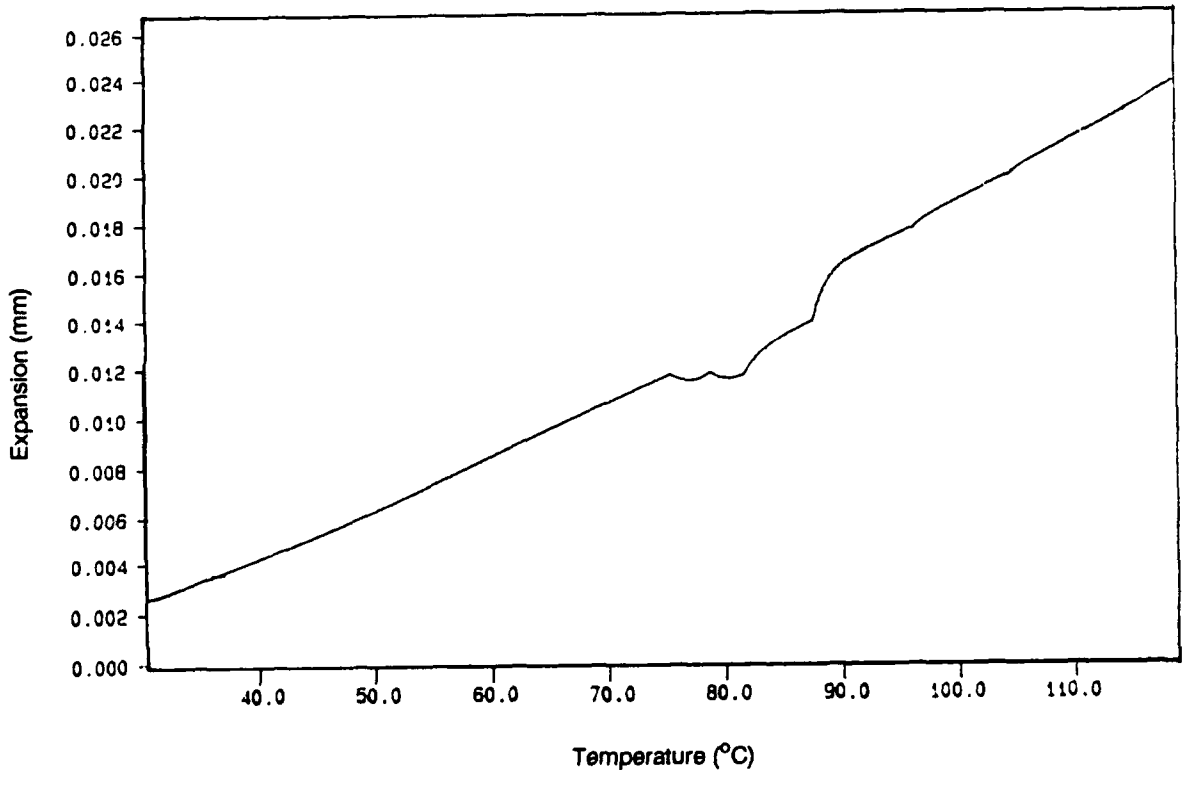

Figure 3b. The thermal expansion for a S-2 glass (8.8 oz.) polyester resin matrix composite perpendicular to the fibers.

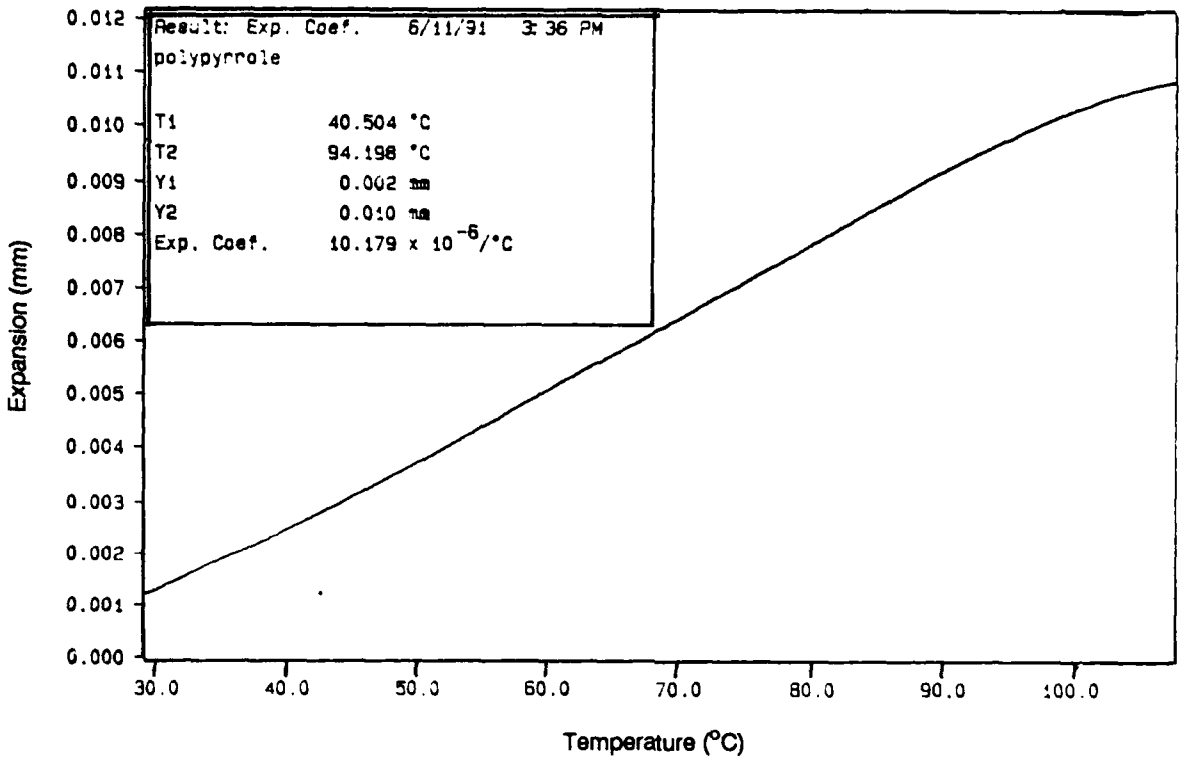

Figure 4. The thermal expansion for a polypyrrole treated S-2 glass (8.8 oz.) polyester resin matrix composite (parallel to fibers).

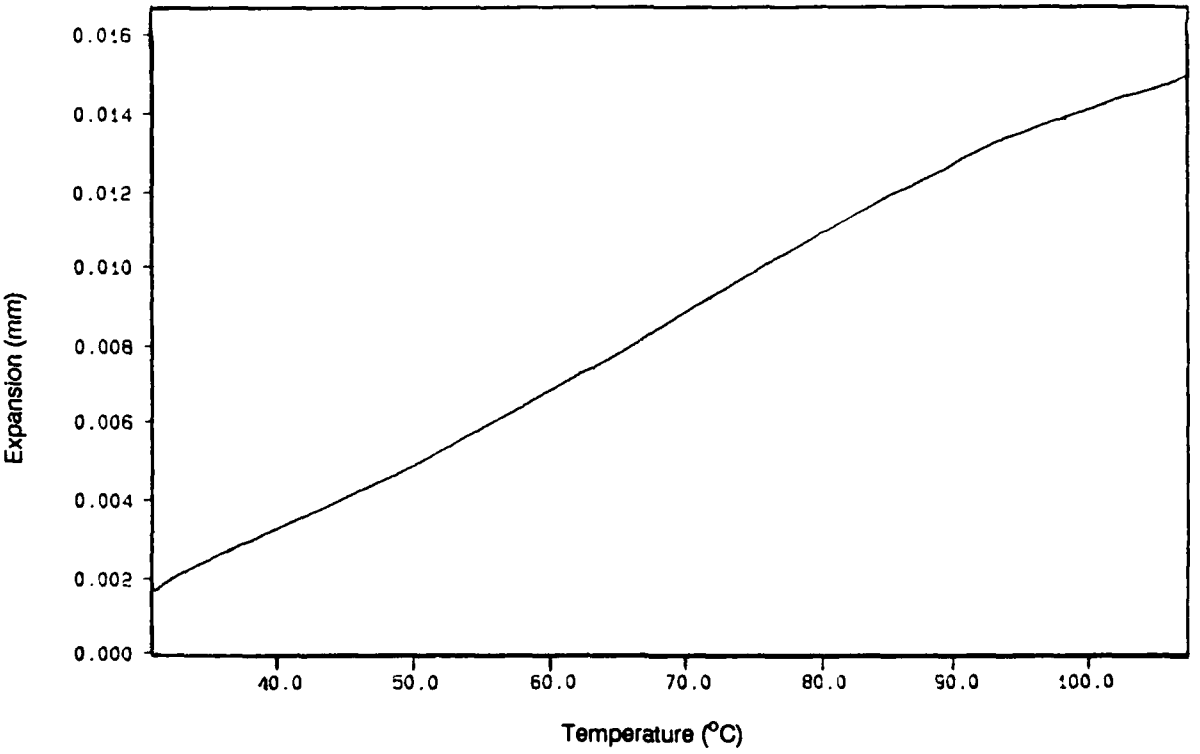

Figure 5a. The thermal expansion for an E-glass polyester resin matrix composite parallel to the fibers.

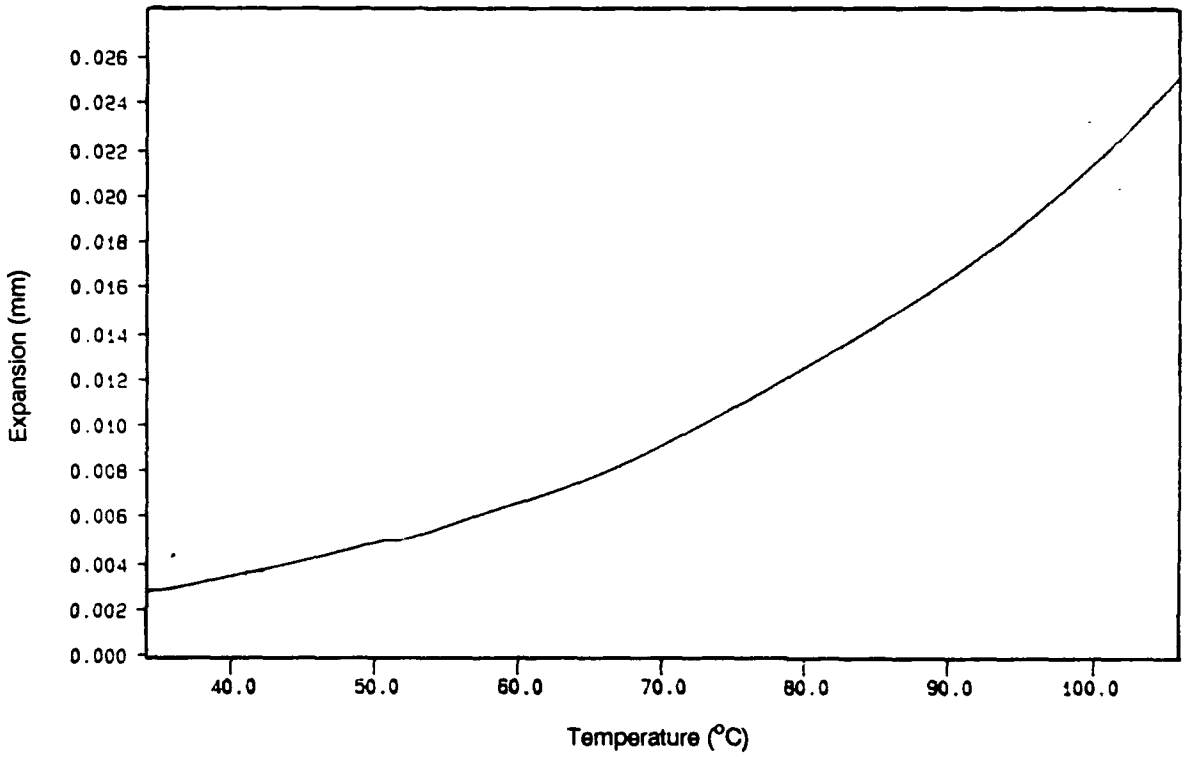

Figure 5b. The thermal expansion for an E-glass polyester

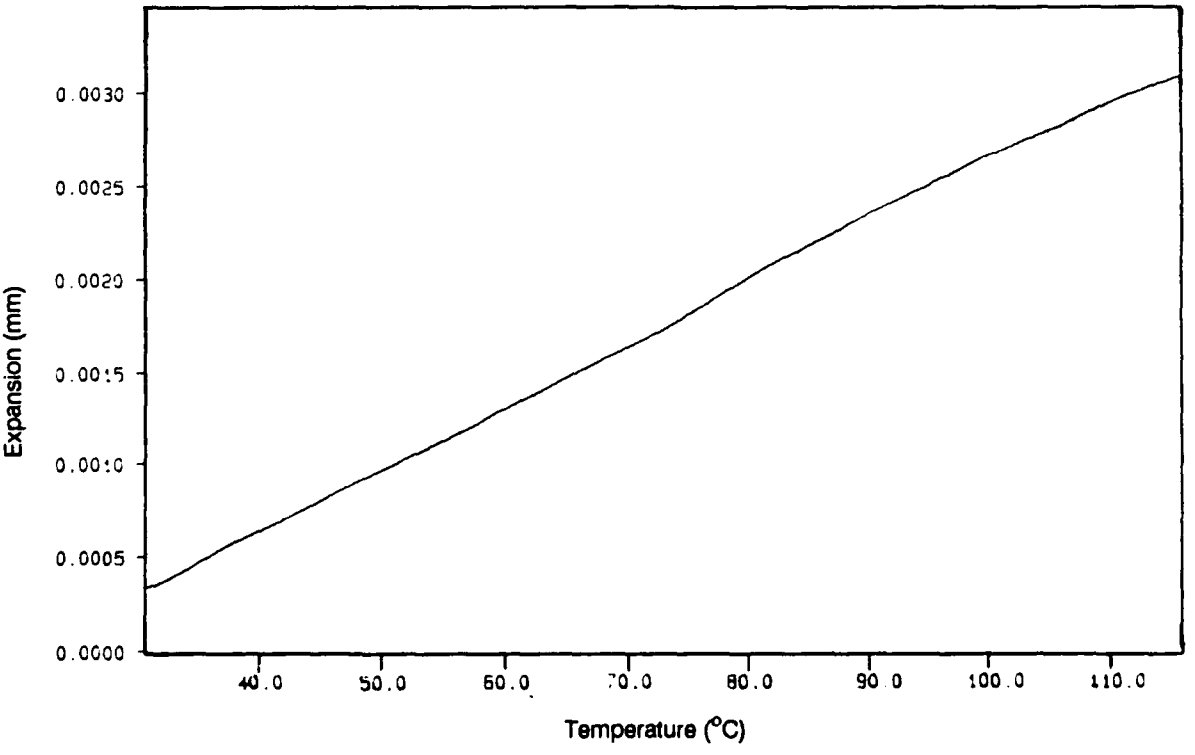

Figure 6a. The thermal expansion for a S-2 glass 5 x 5 (24 oz.)

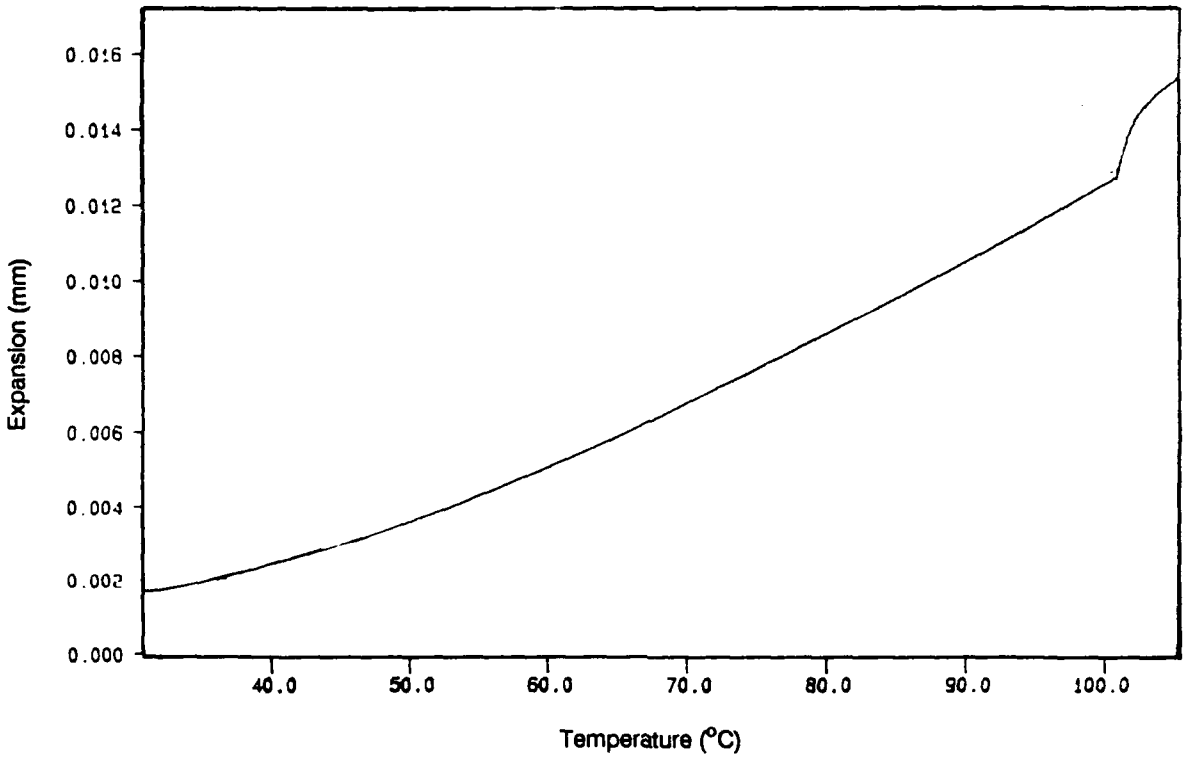

Figure 6b. The thermal expansion for a S-2 glass 5 x 5 (24 oz.) polyester

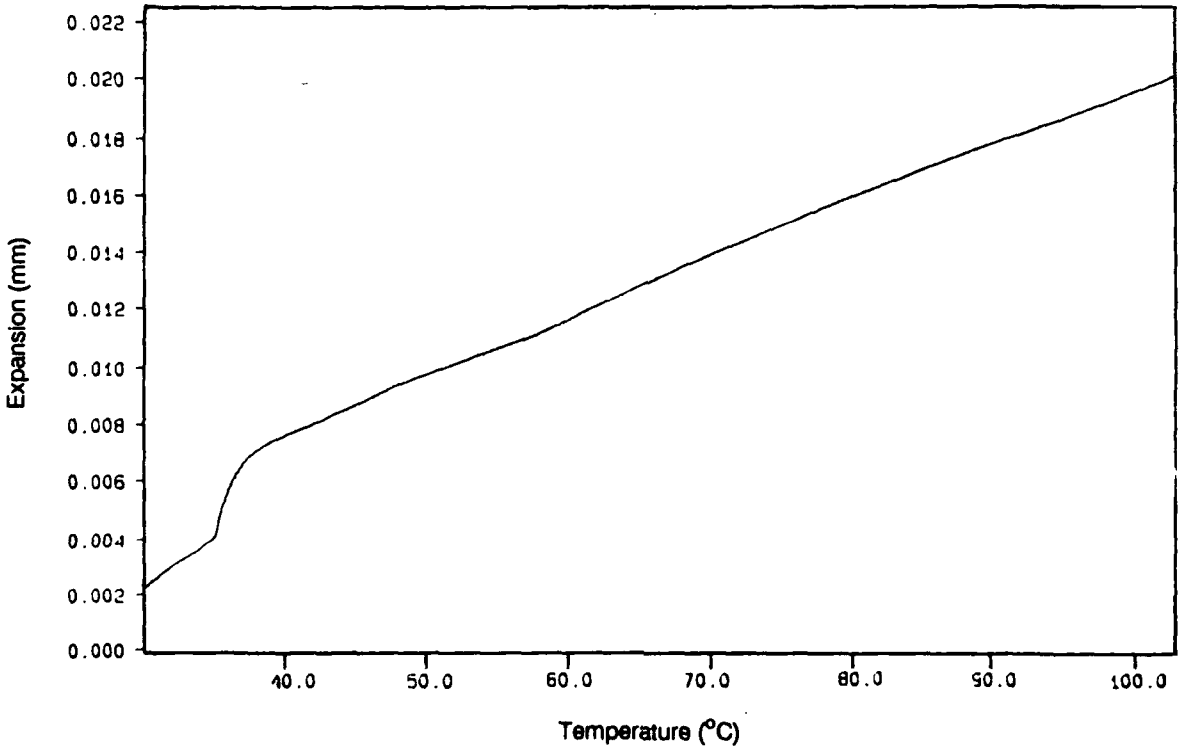

Figure 7a. The thermal expansion for a S-2 glass 3 x 1 (27 oz.) polyester

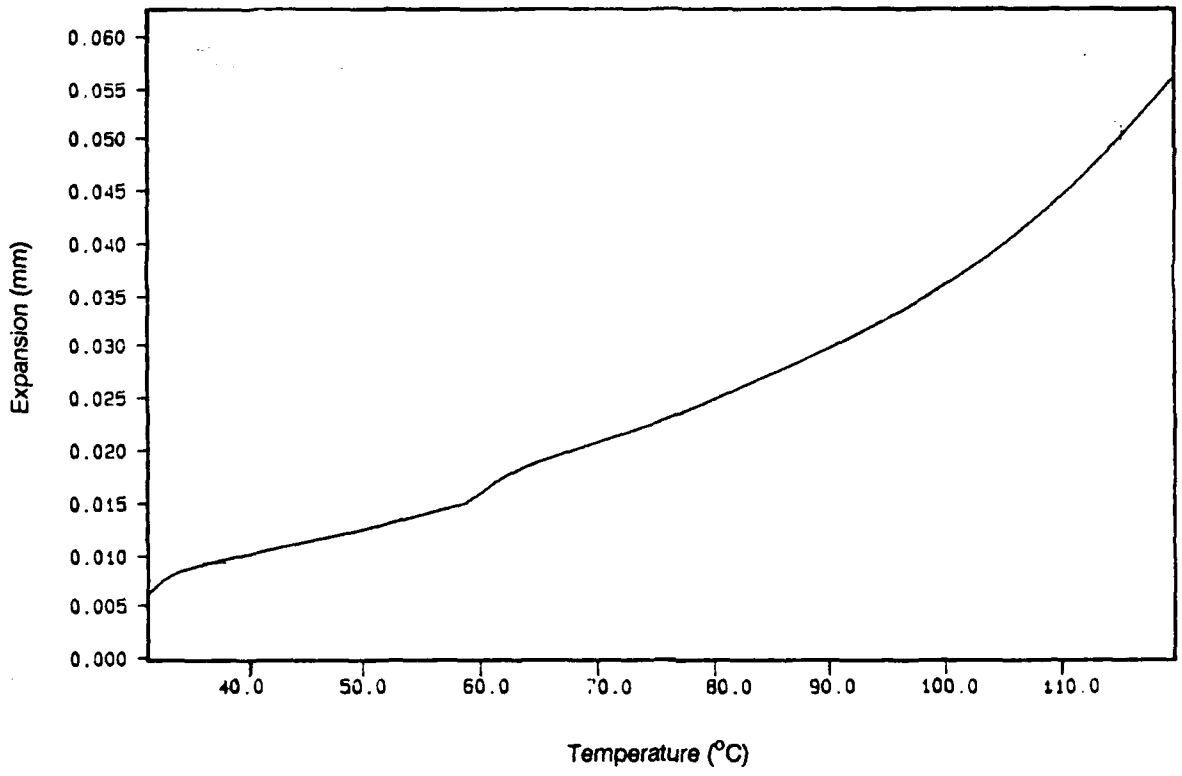

Figure 7b. The thermal expansion for a S-2 glass 3 x 2 (27 oz.) polyester resin matrix composite perpendicular to the fibers.

The fiber volume filling fraction is somewhat smaller for the E-glass and 27 oz. S-2 glass composites. It is also observed the CTEs for the E-glass and S-2 glass 27 oz. composites were higher than those measured for the 8.8 oz. S-2 and the 24 oz. S-2 glass composites; therefore, this behavior is consistent with Equation 2 above.

The composites consistently show an erratic behavior in the expansion coefficient when measured parallel to the fibers. A representative result is shown in Figure 8 for the S-2 glass 5 x 5 (24 oz.) composite. This type of behavior may be caused by small voids which are present in the lamination. The behavior is eliminated or lessened when the samples are annealed under an applied force; however, annealing in an oven without an applied load and then subsequently acquiring the data does not eliminate this behavior as readily. The results reported in Table 3 were all taken after this conditioning treatment.

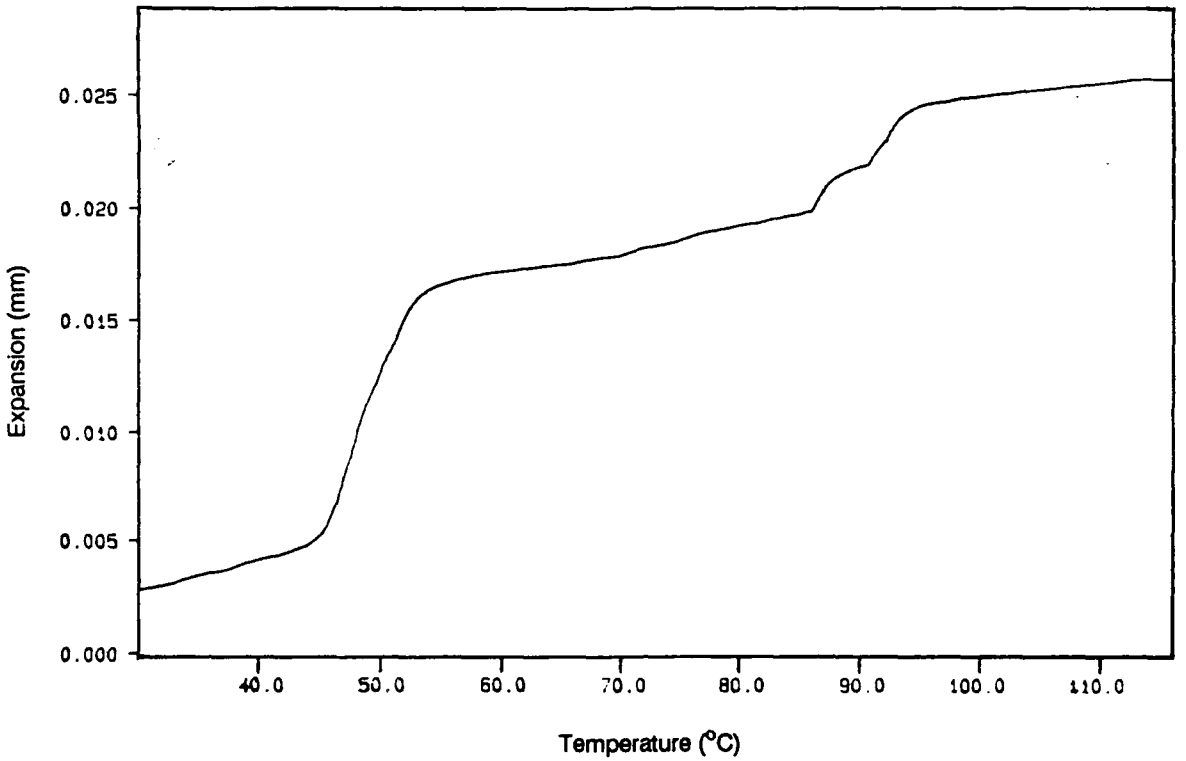

Figure 8. The thermal expansion for S-2 glass composite showing a very nonlinear behavior caused by the inclusion of voids in the laminate.

CONCLUDING REMARKS

The coefficients of thermal expansion for polyester resin, polyester syntactic, epoxy and epoxy syntactic, and glass-polyester resin matrix composites have been determined using a thermal mechanical analyzer. The thermal behavior for the polymers is generally nonlinear and the epoxy resin and syntactic composites show an exaggerated nonlinearity above 60°C. The thermal expansion for the composites is related to the volume filling fraction of the fibers and increases when measured perpendicular to the fibers. An interesting erratic thermal expansion is observed for all the composites when measured parallel to the resin and may be related to voids within the lamination. Further study on the effect of annealing and force on this behavior should be carried out in the future.

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Key Words

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Glass reinforced composites
Coefficients of thermal expansion

Technical Report MTL TR 92-27, April 1992, 14 pp-illus-tables

The coefficients of thermal expansion (CTE) of various polymer matrix materials were measured using a thermal mechanical analyzer. The measurements include a set of standard materials used as calibration materials for the instrument. The data for the base polymers, syntactics, and E and S-2 glass-reinforced polyester matrix composites were obtained in the temperature range from -77°C to 120°C. The results for all of the polymeric materials, especially for the epoxy and epoxy syntactic (above 65°C), are nonlinear. The CTE of the syntactics were reduced by a factor of ~2 for the base resins. The data taken for the glass-reinforced composites show peaks and/or humps in the thermal expansion. These features are believed to be from the formation of voids within the plies of the composite laminate. The irregular behavior of the thermal expansion was minimized by thermal annealing under load conditions, whereas annealing in free-space does not significantly dampen this behavior. The thermal expansion of the material appears to be directly related to the fiber loading.

U.S. Army Materials Technology Laboratory
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THERMAL EXPANSION MEASUREMENTS OF POLYMER MATRIX COMPOSITES AND SYNTACTICS -
Louise C. Sengupta and William A. Spurgeon

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